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DSC (DIFFERENTIAL SCANNING CALORIMETRY) AND HIGH  
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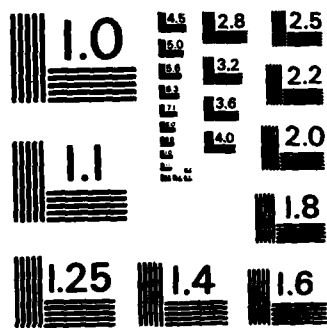
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DSC and High Pressure Conductivity and Electrical Relaxation Measurements  
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by  
John J. Fontanella and Mary C. Wintersgill  
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DSC AND HIGH PRESSURE CONDUCTIVITY AND ELECTRICAL RELAXATION MEASUREMENTS IN PPO AND PPO COMPLEXED WITH LITHIUM SALTS\*

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Differential scanning calorimetry and high pressure electrical conductivity studies have been carried out on poly(propylene oxide) after the addition of various lithium salts. In addition, high pressure electrical relaxation measurements have been performed in the glass transition region on uncomplexed PPO. The vacuum conductivity measurements show that  $T_g$  is 30–40°C lower than the DSC  $T_g$ . Next, it is found that both  $T_g$  and  $T_\infty$  increase several K/kbar. In terms of the configurational entropy model, the isobaric data yield a reasonable value for the activation volume which is consistent with values previously reported for PEO complexed with alkali metal ions. The isothermal data yield a negative activation volume for PPO complexed with LiSCN. Finally, DSC and isothermal conductivity measurements show that neither  $T_g$  nor the apparent activation volume scale directly with ion size.

## 1. INTRODUCTION

Poly(propylene oxide) (PPO) has a  $T_g$  comparable to that of PEO and there have been several studies to determine its complexing properties.<sup>1–4</sup> It is significant that highly amorphous PPO can be prepared. This is particularly important when considering the effect of pressure on the ionic conductivity. Previous work on the pressure dependence of the conductivity of PEO complexed with various salts, particularly the evaluation of the activation volumes, was complicated by the existence of different phases in equilibrium with one another.<sup>5</sup> In the present work, the electrical conductivity of samples of amorphous PPO complexed with several Li salts was studied at high pressure. Also, electrical relaxation and differential scanning calorimetry (DSC) work were carried out.

## 2. EXPERIMENT

PPO in the form of PAREL elastomer was obtained from Hercules, Inc. All of the sample preparation procedures were carried out in a dry box or vacuum oven. The polymer and salts,  $\text{LiCF}_3\text{SO}_3$ ,  $\text{LiClO}_4$ , and  $\text{LiSCN}$  were dissolved in

methanol at about 55°C and cast onto a teflon plate in a glass retaining ring. The samples were initially dried at room temperature in flowing dry nitrogen and then transferred to a vacuum oven for final drying at about 65°C.

The electrical measurements were carried out using techniques described elsewhere.<sup>5</sup> Two terminal high pressure measurements were performed using Spinesstic 22 as the pressure fluid and an oil bath was used for temperature control. Isobaric measurements were performed with about 30 min. equilibration time while for isothermal data the equilibration time was 10 min. DSC was performed with a DuPont 990 DSC interfaced with a microcomputer.

## 3. RESULTS AND DISCUSSION

The conductivity data were analysed using standard complex impedance techniques allowing the determination of the bulk resistance as a function of temperature. A typical impedance plot is shown in fig. 1. These values were used, in conjunction with geometrical measurements, to calculate the bulk conductivity. Neither thermal expansion nor compressibility is included in the data analysis. The results

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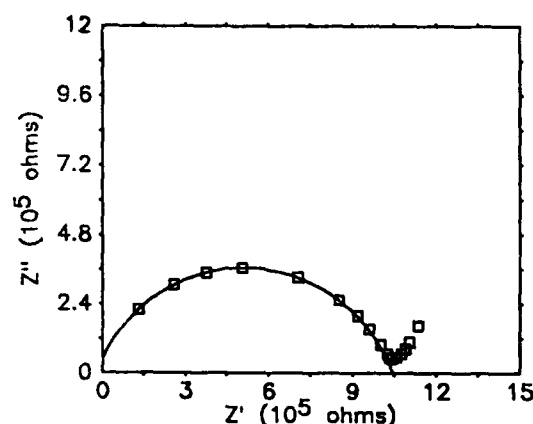


FIGURE 1  
Complex impedance plot for  $\text{PPO}_8\text{LiCF}_3\text{SO}_3$  at 308K and 0.1 GPa.

of typical isobaric data runs are shown in fig. 2. The curvature often observed for amorphous polymer systems is obvious. That the samples were amorphous is confirmed by the DSC results shown in fig. 3. Consequently, the conductivity data were analyzed via the VTF eq.:<sup>6</sup>

$$\sigma = AT^{-1/2} \exp[-E_a/k(T-T_0)] \quad (1)$$

with the adjustable parameters  $A$ ,  $E_a$ , and  $T_0$ . A least squares fit of eq. 1 was carried out and Table 1 contains the best-fit parameters.

Fig. 2 shows the results of these fitting procedures for  $\text{PPO}_8\text{LiCF}_3\text{SO}_3$  at three pressures. The glass transition temperatures,  $T_g$ , obtained from the DSC results shown in fig. 3, are also listed in Table 1.  $T_g$  was obtained by standard straight line analysis. The estimated uncertainties are  $T_g = \pm 5$  K,  $E_a = \pm 0.005$  eV, and  $\log_{10} A = \pm 0.3$ . While the uncertainties are large, some trends can be discerned.

First, the vacuum value for  $T_0$  is always 30–40°C lower than  $T_g$ . This is reassuring since  $T_g - T_0$  is usually on the order of 50°C for polymer systems<sup>7-9</sup> and this is consistent with the configurational entropy model (CEM) of Gibbs and co-workers.<sup>10,11</sup> This considers the temperature variation of the size of the cooperatively rearranging region of the amorphous material in which ion transport occurs and identifies  $T_0$  as the temperature at

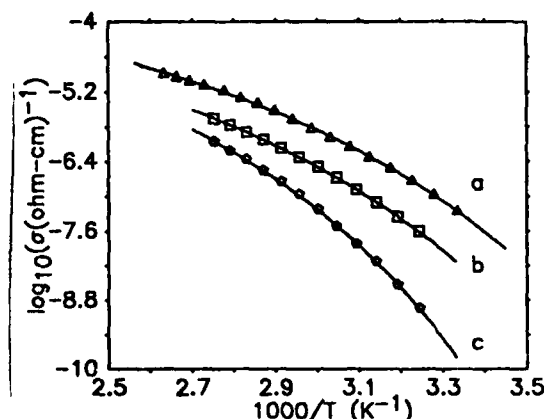


FIGURE 2  
Electrical conductivity vs. reciprocal temperature for  $\text{PPO}_8\text{LiCF}_3\text{SO}_3$  at three pressures: (a) vacuum; (b) 0.1 GPa (1kbar); (c) 0.2 GPa.

which the configurational entropy of the whole system becomes very small.<sup>7,8</sup>

The second trend is that both  $E_a$  and  $T_0$  increase with pressure. Since both quantities increase, this is unlikely to be an artifact of the fitting procedure, since in that case one parameter would tend to increase at the expense of the other. A typical example of this behavior is shown in Table 1 where the results for two samples of  $\text{PPO}_8\text{LiCF}_3\text{SO}_3$  are given. Also, the difference between the present results and that of Armand et al.<sup>1</sup> exhibits this effect.

The result that  $T_0$  increases several K/kbar is consistent with the contention that  $T_0$  is somehow related to the glass transition, since  $T_g$  normally increases by this amount. Some evidence for this behavior in uncomplexed PPO is given in fig. 4 where electrical relaxation results are shown. That relaxation is associated with the glass transition and the shift in the maximum temperature,  $T_m$ , is about 17 K/kbar which is consistent with the above result. Unfortunately, similar measurements cannot be carried out on complexed materials because of the high DC conductivity. DSC measurements under pressure which would yield this information are currently being undertaken. Still other evidence that  $T_0$  is related

TABLE 1. DSC results for  $T_g$  and best fit parameters in equations 1, 5, and 6.

Salt in PPO (8:1)	RMS Deviation	$\log_{10} A$	$E_a$ (eV)	$T_o$ (K)	$T_g$ (K)	$C_1$	$C_2$ (K)	$\log_{10} \sigma(T_g)$	$\log_{10} A'$	$E_a'$ (eV)	$T_o'$ (K)
<b>LiCF<sub>3</sub>SO<sub>3</sub></b>											
Vacuum #1	0.021	-0.49	0.092	210	238	16.9	25.9	-18.8	-1.90	0.087	212
Vacuum #2	0.020	-0.94	0.086	214	238	18.7	22.0	-21.0	-2.34	0.082	216
0.1 GPa	0.0063	-1.02	0.098	216	255	12.6	37.1	-15.1	-2.41	0.093	218
0.2 GPa	0.016	-0.73	0.104	233	272	13.3	38.1	-15.4	-2.11	0.101	234
<b>LiClO<sub>4</sub></b>											
Vacuum	0.013	-0.04	0.081	235	263	14.8	26.4	-16.2	-1.44	0.077	237
<b>LiSCN</b>											
Vacuum	0.011	+0.047	0.126	203	245	15.2	39.9	-16.1	-0.95	0.120	205

to  $T_g$  is that PPO<sub>8</sub> LiClO<sub>4</sub> gave rise to a value of 235 K for  $T_o$ . This is significantly higher than any of the results for lithium triflate or thiocyanate, and is consistent with the DSC results for  $T_g$ . Also, Armand et al.<sup>1</sup> have shown that  $T_o$  increases with increasing salt concentration as is observed for  $T_g$ .<sup>12</sup>

Using the CEM, the shift in  $E_a$  can be used to calculate the activation volume,  $v^*$ . With the usual assumptions,<sup>5</sup> it follows that:

$$\frac{1}{E_a} \frac{dE_a}{dP} = \frac{1}{T_o} \frac{dT_o}{dP} + \frac{v^*}{g} \quad (2)$$

yields  $v^*/g = 37 \pm 40$  cm<sup>3</sup>/mol-eV for PPO<sub>8</sub>:LiCF<sub>3</sub>SO<sub>3</sub>. This value is the difference between two large, relatively uncertain quantities, however the mean value is reasonable since, for a value of  $g=1$  eV, for example, the activation volume is similar to the values quoted previously for alkali metals in PEO.<sup>5</sup> Consequently, the comments made for PEO concerning the transport mechanism<sup>5</sup> also hold for PPO.

In order to compare the effects of pressure on PPO complexed with the various salts, isothermal data were also obtained. Linear least square best fits were obtained and the results at 50°, 70° and 90°C are shown in Table 2. The 70°C data and best fit curves are shown in fig. 5. Also, the values for an "Arrhenius" activation volume were calculated from:

$$v_{Arr}^* = -kT \, d \ln g / dP \quad (3)$$

and are also listed in Table 2. Finally, values for  $v^*/g$  were calculated using:

$$\frac{v^*}{g} = \frac{(T-T_o)}{K_\sigma} \left[ \frac{d \ln A}{dP} - \frac{d \ln \sigma}{dP} \right] - \frac{T}{T_o(T-T_o)} \left( \frac{dT_o}{dP} \right) \quad (4)$$

where the assumptions for all materials are that  $d \ln A / dP = 0$  and that  $dT_o / dP = 9.9$  K/GPa, which was observed for PPO<sub>8</sub>:LiCF<sub>3</sub>SO<sub>3</sub>. The remaining parameters are those listed in Tables 1 and 2.

The pressure variation of the conductivity for PPO<sub>8</sub>:LiClO<sub>4</sub> is consistently larger than for PPO<sub>8</sub>:LiCF<sub>3</sub>SO<sub>3</sub> which, in turn, is larger than that for PPO<sub>8</sub>:LiSCN. Since triflate is the largest anion, the slope does not scale with the size of the anion. Similar measurements on PEO containing LiClO<sub>4</sub> and LiSCN were the basis for the suggestion that the slope scales with ion size.<sup>5</sup> However, LiCF<sub>3</sub>SO<sub>3</sub> was not studied in that case. In the PEO work it was pointed out that the simplest explanation of the ion size effects was that the anions were mobile. That explanation could still hold if a larger fraction of the current is carried by Li ions in the case of LiCF<sub>3</sub>SO<sub>3</sub> complexed material. However, as was also pointed out in the PEO work, it may be that other effects such as morphology changes may be the explanation.

$T_g$  also shows the same general trend as the effect of pressure on the conductivity in that



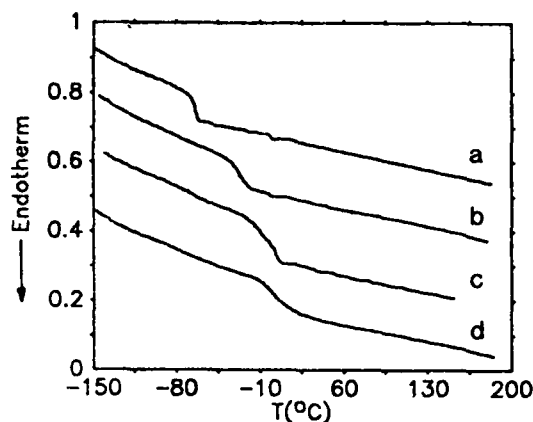


FIGURE 3

DSC thermograms for: (a) uncomplexed PPO; (b)  $\text{PPO}_8\text{LiCF}_3\text{SO}_3$ ; (c)  $\text{PPO}_8\text{LiSCN}$ ; (d)  $\text{PPO}_8\text{LiClO}_4$ .

$\text{LiClO}_4$  exhibits the highest  $T_g$ . A possible explanation for these effects may be related to the fact that both the thiocyanate and triflate ions have a dipole moment, in contrast to the perchlorate ion which does not. For example the added dipole interaction could lead to a configuration which decreases both  $T_g$  and the effect of pressure on the conductivity. It will be of interest to study PPO complexed with other lithium salts to test this idea.

Next, the value of  $v^*/g$  listed in Table 2 for  $\text{PPO}_8\text{LiSCN}$  is negative. This result is reminiscent of an observation for PEO.<sup>5</sup> That was considered an anomaly primarily due to an improper choice of  $T_0$ . That is obviously not the explanation in the present work and presumably not for PEO either. There are several assumptions in the calculations based on eq. (4) and thus this result is not definitive. Some of those assumptions are eliminated via isobaric data and such experiments are currently underway.

Finally, in the  $\ln(G)$  vs  $P$  plots for PPO, there appears to be a small curvature at lower temperatures which is opposite in sense from that reported for PEO. The effects of this curvature are also apparent in fig. 2. It is likely that the different curvature for the PEO plots arises because the PEO results were

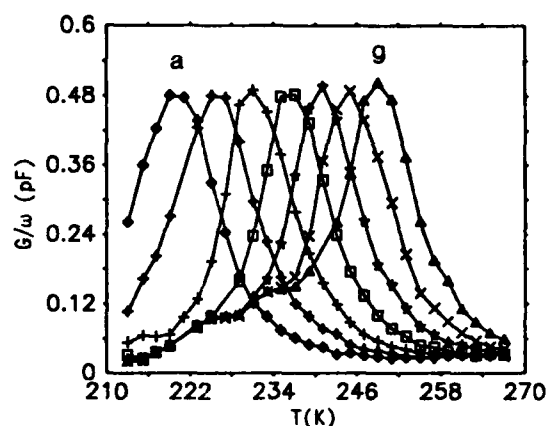


FIGURE 4

Conductance vs. temperature for uncomplexed PPO at various pressures. The curves from left to right are: (a) 100 MPa (1 atm); (b) 0.03 GPa; (c) 0.06 GPa; (d) 0.09 GPa; (e) 0.12 GPa; (f) 0.15 GPa; (g) 0.18 GPa.

single frequency data rather than the result of complex impedance analysis. In fact, a decrease in the curvature with frequency is obvious from fig. 1 of that paper. Presumably, the downward curvature at lower temperatures in PPO is due to the shift of  $T_g$  with pressure.

For comparison, the data were reanalyzed in terms of the WLF equation:<sup>13</sup>

$$\log_{10} \frac{\sigma(T)}{\sigma(T_g)} = \frac{C_1(T-T_g)}{C_2+(T-T_g)} \quad (5)$$

The resultant parameters are listed in Table 1. The values of  $C_1$  and  $C_2$  are as close to the "universal" values of 17.4 and 51.6 as are those of Watanabe et al.<sup>2</sup>

Finally, for completeness, the data were analyzed via the VTF eq. in the form:

$$\sigma = A' \exp\left[-\frac{E_a'}{(T-T_0')}\right] \quad (6)$$

The results are also listed in Table 1.

#### 4. CONCLUSIONS

In summary, then, the effect of pressure on the electrical conductivity for PPO complexed with lithium salts has been determined and DSC measurements have been carried out. In

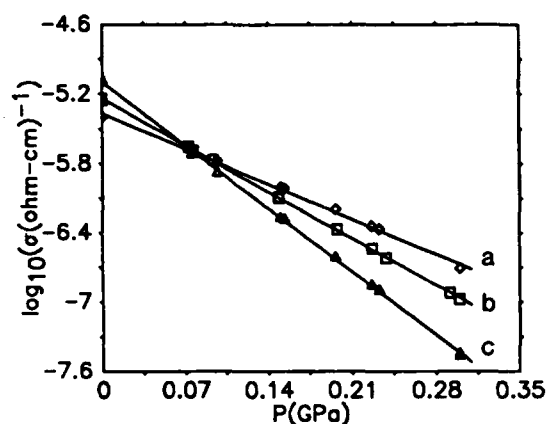


FIGURE 5  
Conductivity vs. pressure for: (a) PPOgLiSCN;  
(b) PPOgLiCF<sub>3</sub>SO<sub>3</sub>; (c) PPOgLiClO<sub>4</sub>.

addition, high pressure electrical relaxation measurements have been performed in the glass transition region on uncomplexed PPO. The vacuum conductivity measurements show that  $T_0$  is 30–40°C lower than the DSC  $T_g$ . Next, both  $T_0$  and  $T_g$  increase several K/kbar. Also, the isobaric data yield a reasonable value for the activation volume which is consistent with values previously reported for PEO complexed with alkali metal ions. The isothermal data yield a negative activation volume for PPOg-LiSCN. Finally, it is found that neither  $T_g$  nor the Arrhenius activation volume scales directly with ion size.

#### ACKNOWLEDGMENTS

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TABLE 2. Pressure variation of the conductivity and activation volumes for PPO complexed with lithium salts.

Salt in PPO (8:1)	T (°C)	$\frac{d \ln \sigma}{dP}$ (GPa) <sup>-1</sup>	$v^*/g$ (cm <sup>3</sup> /mol-eV)	$v_{Arr}^*$ (cm <sup>3</sup> /mol)
LiClO <sub>4</sub>	50	-24.0	68	64
	70	-17.0	60	58
	90	-14.2	72	43
LiCF <sub>3</sub> SO <sub>3</sub>	50	-18.9	67	51
	70	-13.3	47	38
	90	-10.5	41	32
LiSCN	50	-13.0	-23	35
	70	-10.0	-23	29
	90	-9.1	-11	24

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